FIFTH MONTHLY PROGRESS REPORT ON DEVELOPMENT AND TESTING OF ELECTROLYTE 9N64-18284* MATRIX COMBINATIONS FOR MERCURY-POTASSIUM FUEL CELL (12 Apr - 12 May 1963)CONTRACT NASw-476 Date Submitted: 25 May 1963 V. L. Decker Project Manager Research Activity Director of Research Allison Division 1016732 General Motors Corporation Indianapolis, Indiana

PROGRESS OF WORK DURING THIS REPORTING PERIOD

The time-phased chart shown in Figure 1 has been modified to reflect the latest adjustments in the program. It reflects an increase in the time requirement for preparation, strength testing, and conductivity testing of both the coarse and fine grain composite electrolytes. These delays are explained under the specific task reports. A composite with 60% electrolyte was prepared from the fine grain. Conductivity was measured near 0.3 mho/cm at cell operating conditions.

A milestone was placed on the original schedule (Figure 1) mid-March, in anticipation of seal problems with the first small cells. However, these small cells displayed good performance for a short period of time (reference Fourth Monthly Progress Report, Allison EDR 3314). Failure was not directly attributed to the seal of the composite to the cell. Quality control studies, reported under Task II of this report, show that additional effort will be required in the development of preparation and/or handling techniques. This should be done before a large composite disk can be selected for large cell fabrication.

More detailed data on progress for this period is reported as follows by task.

TASK I

Coarse Grain Composite

A new composite electrolyte batch was made up to contain 34% electrolyte—the composition was found to be inaccurate. Therefore, another nominal 34% composite batch was made. The exact analysis is being checked. Physical test data on this batch shows strength values of 2.75 psi at 350°C, using an 87% of theoretical density specimen. Flowability tests at 350°C reveal that the material undergoes at least a 20% linear deformation before cracks appear.

Fine Grain Composite

Strength and flowability tests have been completed on specimens made from 55%, 57.5%, and 60% electrolyte. The data are summarized as follows:

- Strength values increase with temperature in the range from 250 to 350°C, and decrease with higher electrolyte content. It has always been predicted that the strength will drop with higher saturation caused by the increase in electrolyte content, but the increase with temperature is unexpected and warrants further analysis.
- Flowability values decrease with an increase in temperature, but show the characteristic increase with higher saturation.

This preliminary data shows a reverse order for properties with temperature, and appears to show a surface strength associated with bake-out. Surface strength would show up in this manner on both tests. Blistering of the surface from apparent gassing during preparation has been observed on samples made from these batches. A recheck of the procedure and the materials used will be made.

TASK II

Conductivity Measurements

Nine conductivity specimens were made up using the fine grain composite—three each of the 55, 57.5, and 60% electrolyte content batches. A comparison of the fine grain to the coarse grain results shows about a factor of three improvement in conductivity at 300°C. The coarse grain averaged out at approximately 0.1 mho/cm, while the value for the fine grain is near 0.3 mho/cm.

Further testing of the 34% electrolyte composites was not accomplished because the material was not ready for this work.

Small Cell Testing

No new small cell testing has been done. The 34% composite was not completed, and a 60% fine grain composite fractured prior to delivery for cell testing.

Quality Control Studies

Compatibility studies were initiated to obtain some insight into the quality control on the composite matrix materials. A number of tests were completed—some were severe, but informative. The tests are listed below with (1) conditions, (2) results, and (3) conclusions (coarse grain composites were used in all tests).

• Composite in Mercury

- 1. 300°C, 24 hours
- 2. Some gassing from the specimen, no penetration of metal into composite-geometry remained the same
- 3. No problem with Hg, however, gas liberation must be investigated

Composite As-received

- 1. Broken at room temperature under mineral oil
- 2. Gas bubbles observed by scope coming from pockets or cracks within the material
- 3. Should be prepared under vacuum

Composite in Potassium

- 1. 300°C, 24 hours
- 2. Composite dissolved in liquid metal
- 3. The electrolyte is known to be soluble in potassium, which varifies that a strong mechanism is set up to remove the electrolyte and destroy the composite structure

- Composite in 50-50% Molar K-Hg Amalgam
 - 1. 300°C, 42 minutes
 - 2. Composite was seen to gas and breakup
 - 3. The presence of Hg does not alter the solubility of electrolyte into the potassium as a mechanism for destruction of the material
- Composite in Electrolyte-saturated Potassium
 - 1. 35% composite supported over liquid metal prior to immersion into the pool
 - 2. Composite became soft, deforming over the supporting wire harness, and parting to fall into the pool
 - 3. Shows the weakness of this material which has displayed 50% deformation in flowability tests
- Composite in Electrolyte-saturated Potassium
 - 1. 300°C, 24 hours—composite carefully prepared for test with low density and vacuum baked
 - 2. Observed to remain intact during test. Post-test cleaning showed cracks formed. Breaking along one crack showed that severe attack had taken place throughout, and the two parts were held by less than 10% of the original composite material. In this region, the degree of potassium penetration was low and spotty. Other work has also shown this nonuniform composite structure.
 - 3. Composite would not have survived a long run under cell operating conditions

These tests indicated that the present technique for composite preparation and handling is not satisfactory. Additional work will be required in the following areas:

- 1. Material degassing
- 2. Elimination of temperature cycling to reduce internal stress
- 3. Other material refinements found necessary

WORK FOR NEXT REPORTING PERIOD

TASK I

The development program for preparation techniques is being redirected, as indicated in Figure 1. Emphasis will be placed on a critical review of the presently stated procedures in an effort to eliminate moisture and gas pickup and nonuniform grain and salt distribution by the material. The program will be flexible to allow for changes, as newly devised tests indicate the need. Equipment and additional experienced personnel will be transferred to the program as needed.

Two specific areas to be completed next report period are:

- 1. Complete the evaluation of both high and low density specimens using the newly prepared 34% composite
- 2. Prepare a 63% fine grain electrolyte composite and determine the maximum electrolyte content at which compressive strength is still detectable

TASK II

Small cell testing will be used to test material if other less complex tests are not thought to suffice. Compatibility tests will continue to be the main-stay of the quality control program, with new tests being introduced as needed.

Conductivity tests will be performed on specimens from the 34% batch recently prepared. Also, the conductivity of a newly prepared electrolyte will be established for later correlation with the composite conductivity study.

TASK III

Work on the design of large cells will be dependent on information from Tasks I and II.

CUMULATIVE MAN MONTHS EXPENDED

- RESEARCH-13.2 Man Months, a balance of 10.8 Man Months for the remainder of the program
- SHOP-0.8 Man Months, a balance of 1.2 Man Months for the remainder of the program
- MATERIALS LABORATORY-7.8 Man Months, a balance of 4.2 Man

 Months for the remainder of the program

ORIGINAL BUDGET

- Research-24.0 Man Months
- Shop—2.0 Man Months
- Materials Laboratory-12.0 Man Months